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Indian Standard

SPECIFICATION FOR
4,4′-DIAMINOSTILBENE-2,2′-DISULPHONIC ACID

(First Revision)

UDC 667·21·547·425·5

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Indian Standard

SPECIFICATION FOR 4,4′-DIAMINOSTILBENE-2,2′-DISULPHONIC ACID

(First Revision)

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Indian Standard

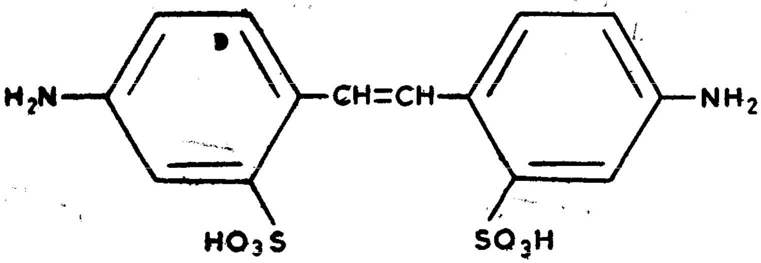
SPECIFICATION FOR 4,4'-DIAMINOSTILBENE-2,2'-DISULPHONIC ACID

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 31 July 1975, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 4,4'-Diaminostilbene-2,2'-disulphonic acid ($C_{14}H_{14}O_6S_2N_2$) is an intermediate widely used in the manufacture of dyes as well as for optical brightening agents. It has the following structural formula:



4,4'-Diaminostilbene-2,2'-Disulphonic Acid
(Molecular Mass 370·4)

0.3 Diaminostilbene disulphonic acid is supplied both as disodium salt and as free acid. It was, therefore, felt by the Sectional Committee responsible for the preparation of this standard, that the revised version should include the specifications for both disodium salt and the free acid. Two minimum values of assay have, therefore, been included in this revision.

0.4 Technical grade of diaminostilbene disulphonic acid contains a number of impurities, three of which have so far been identified, namely, 4,4'-diaminodibenzyl-2,2'-disulphonic acid, 4-aminobenzaldehyde and 4-aminotoluenesulphonic acid. Of the three, 4,4'-diaminodibenzyl-2,2'-disulphonic acid is most objectionable, especially if the material is to be used for manufacture of optical whitening agents. Limit of this impurity and method of its estimation has also been included in this revision.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 4,4′-diaminostilbene-2,2′-disulphonic acid which may be in the form of free acid or disodium salt.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a paste or in the form of yellowish or yellowish-brown needles, lumps or powder.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 4,4′-DIAMINOSTILBENE-2,2′-DISULPHONIC ACID

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Purity, as percent nitrite value (on dry basis), *Min*:		
	a) As free acid (molecular mass 370·4)	95	A-2
	b) As disodium salt (molecular mass 414·4)	70	A-2
ii)	Insolubles in ammonia solution, percent by mass, *Max*	0·25	A-3
iii)	4,4′-Diaminodibenzyl-2,2′-disulphonic acid, percent by mass (on dry basis), *Max**	1·0	A-4

*This requirement is applicable only when the material is to be used for the manufacture of optical whitening agents.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1970†) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

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*Rules for rounding off numerical values (*revised*).

†Specification for steel drums (galvanized and ungalvanized) (*first revision*).

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Percentage purity of the material;
c) Name of the manufacturer and his recognized trade-mark, if any;
d) Gross, net and tare mass; and
e) Batch number.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. The ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for the determination of purity shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of remaining characteristics, namely, insolubles in ammonia solution and 4,4′-diaminodibenzyl-2,2′-disulphonic acid shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of purity if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

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*Methods of sampling and tests for dye intermediates.

5. TEST METHODS

5.1 Tests shall be carried out according to methods prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

PPENDIX A

(*Clauses* 2.2 *and* 5.1)

METHODS OF TEST FOR 4,4′-DIAMINOSTILBENE-2,2′-DISULPHONIC ACID

A-1. PREPARED SAMPLE

A-1.1 If the sample is in the form of a powder, mix well by rotating the bottle several times. If the sample is in the form of a paste, stir it well. Dry at 110°C to constant mass. Cool in a desiccator and transfer the dry sample to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. *Use this prepared sample for tests.*

A-2. DETERMINATION OF PURITY

A-2.0 Outline of the Method — The material is dissolved in aqueous ammonium hydroxide. A known volume of the solution is diazotized with standard sodium nitrite solution and concentrated hydrochloric acid, and the excess of standard sodium nitrite solution is measured by titration with standard 4-nitroaniline solution.

A-2.1 Reagents

A-2.1.1 *Ammonium Hydroxide Solution* — 10 percent (*v/v*).

A-2.1.2 *Standard Sodium Nitrite Solution* — 0·2 N.

A-2.1.3 *Standard 4-Nitroaniline Solution* — 0·2 N.

A-2.1.4 *Starch-Iodide Test Papers*

A-2.1.5 *Concentrated Hydrochloric Acid* — *See* IS : 265-1962†.

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*Specification for water, distilled quality (*revised*).

†Specification for hydrochloric acid (*revised*).

A-2.2 Procedure — Weigh accurately about 5 g of *the prepared sample* (*see* **A-1.1**). Dissolve this in a 400-ml beaker with the requisite amount of ammonium hydroxide solution and water. Transfer the solution to a 500-ml volumetric flask and make up to the mark with water. Mix well and pipette a 100-ml aliquot of the solution into a one-litre iodine flask. Add 200 ml of water. Then from a burette, add sufficient standard sodium nitrite solution so as to provide not more than 2 to 3 ml excess of the solution (*see* Note). Cool in an ice-bath to about 10°C. Add quickly 20 ml of concentrated hydrochloric acid. Stopper the flask immediately. Mix well and allow to stand in the ice-bath with occasional stirring for a period of 10 minutes. At the end of this period, test for the presence of excess nitrite by spotting on starch iodide test paper. Back titrate excess of standard sodium nitrite with standard 4-nitroaniline solution to just no-blue colour on starch iodide test paper. Titrate the same amounts of water and hydrochloric acid as was present at the end of sample titration, with standard sodium nitrite solution and note this as the reagent blank.

NOTE — To know approximately the volume required, carry out a preliminary titration according to the procedure prescribed.

A-2.3 Calculation

A-2.3.1 Purity, percent nitrite value:

a) Calculated as 4,4′-diaminostilbene-2,2′-disulphonic acid (molecular mass 370·4) $= \dfrac{(V_1 - V_2 - V_3) \times 18{\cdot}5}{M}$

b) Calculated as disodium salt, (molecular mass 414·4) $= \dfrac{(V_1 - V_2 - V_3) \times 20{\cdot}7}{M}$

where

V_1 = volume in ml of sodium nitrite solution added,

V_2 = volume in ml of 4-nitroaniline solution required for back titration,

V_3 = volume in ml of sodium nitrite solution consumed for blank titration, and

M = mass in g of the material taken for the test.

A-3. DETERMINATION OF INSOLUBLES IN AMMONIA SOLUTION

A-3.1 Procedure — Weigh accurately about 10 g of the material. Transfer to a 500-ml beaker. Add slowly ammonium hydroxide solution (10 percent) while stirring with a rod until distinctly alkaline to phenolphthalein test paper. Stir until dissolved. Filter through a tared sintered or Gooch crucible and dry in an oven at 100 ± 2°C. Cool in a desiccator and weigh. Calculate percentage residue.

A-4. DETERMINATION OF 4,4′-DIAMINODIBENZYL-2,2′-DISULPHONIC ACID

A-4.0 Outline of the Method — The estimation of this impurity is done by thin layer chromatography.

A-4.1 Apparatus

A-4.1.1 *Thin Layer Chromatography Plate* — glass plate of size 20 × 20 cm coated uniformly with MN cellulose 300 G or an equivalent powder, which is prepared and applied as follows:

> Mix 30 g of the powder with 160 ml of water to form a homogeneous suspension and apply it to the glass plate in a thickness of 0·25 mm. Dry the plate at room temperature for 30 minutes and then at 110°C for 35 minutes in a drying oven with air circulation.

A-4.1.2 *Micropipette* — 5 microlitre capacity.

A-4.1.3 *Developing Chamber* — suitable rectangular glass jar which contains the mobile phase (developer) and which is closed well with the lid for saturation.

A-4.1.4 *Chromatographic Sprayer*

A-4.2 Reagents

A-4.2.1 *4,4′ - Diaminodibenzyl - 2,2′ - Disulphonic Acid* — pure (reference sample).

A-4.2.2 *Ammonium Hydroxide* — 25 percent (*v/v*).

A-4.2.3 *Acetone* — *See* IS : 170-1966*.

A-4.2.4 *Developer or Mobile Phase* — 20 percent (*m/v*) sodium chloride solution.

A-4.2.5 *Spray Solution A* — Mix 1 N hydrochloric acid and 1 N sodium nitrite solution in the ratio of 2 : 1.

A-4.2.6 *Spray Solution B* — 0·5 percent (*m/v*) solution of H-salt in 1 N sodium hydroxide solution.

A-4.3 Procedure

A-4.3.1 Weigh accurately 2 g of the reference sample in an amber-coloured weighing bottle and dissolve in 100 ml of a solvent mixture of water, ammonium hydroxide and acetone in the ratio of 4 : 1 : 5. From this solution prepare a series of standard solutions numbering 5 to contain known quantity of the sample.

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*Specification for acetone (*first revision*).

A-4.3.2 Using a micropipette, spot 5 microlitre each of all these solutions on to the thin layer plate. Care shall be taken to see that all the spots are in the same line. Similarly prepare the solution of the material under test and make a spot of this also in line with other reference standards on the thin layer plate. Allow the plate to stand for 5 minutes in the dark so that the spots applied dry up. The developer is placed in the chamber which is maintained at 22°C and at a relative humidity of 50 percent. Close the chamber with its lid and allow to achieve equilibrium for 1 hour. Now place the plate carefully in the developer and allow the mobile phase to run in an ascending manner for a distance of about 12 cm from the spots. This will take approximately 1½ to 2 hours. Remove the plate from the chamber and dry it completely. Spray the plate with spray solution *A* followed by spray solution *B*. After a few minutes examine visually the intensity of colour developed with the material under test and compare it with that of the known reference sample.

Report the 4,4′-diaminodibenzyl-2,2′-disulphonic acid content as that which is nearest in intensity to the standard. In case the colour intensity of the test sample does not come within the range of the standard spots, repeat the test using a different percentage of 4,4′-diaminodibenzyl-2,2′-disulphonic acid.

A-4.3.2.1 *Identification* — Details of the chromatogram are given in Table 2.

TABLE 2 CHROMATOGRAM

SL No.	ZONE	R_F VALUE	COLOUR		SUBSTANCE
			(a)	(b)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	1, 2, 3	0	Blue fluorescence	Blue	Unknown
ii)	4	0·38	do	Blue	4,4′-Diaminostilbene-2,2′-disulphonic acid
iii)	5	0·55	do	Violet	Unknown
iv)	6	0·60	—	Red	4,4′-Diaminodibenzyl-2,2′-disulphonic acid
v)	7	0·65	—	Violet	4-Aminobenzal dehyde
vi)	8	0·80	Blue fluorescence	Violet	Unknown
vii)	9	0·85	—	Red	4-Aminotoluene-sulphonic acid

NOTE — ‘a’ denotes fluorescence seen under ultraviolet light.

‘b’ denotes the colour of the zone developed by diazotization and coupling with H-acid.

INDIAN STANDARDS

ON

SULPHONIC ACID DYE INTERMEDIATES

IS :

2740-1973	Sulphanilic acid, technical (*first revision*)
3229-1973	Naphthionic acid (sodium salt) (*first revision*)
4265-1975	4,4′-Diaminostilbene-2,2′-disulphonic acid (*first revision*)
4425-1967	*p*-Nitrotoluene-*o*-sulphonic acid
4528-1968	4,4′-Dinitrostilbene-2,2′-disulphonic acid (disodium salt)
5045-1969	Metanilic acid, technical
5299-1969	Methods of sampling and tests for dye intermediates
5438-1969	Nitrobenzene-*m*-sulphonic acid, sodium salt
6264-1971	J-acid
7362-1974	Tobias acid
7645-1975	Phenyl J-acid, technical
7646-1975	Benzoyl J-acid, technical
7704-1975	4-Aminotoluene-3-sulphonic acid

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